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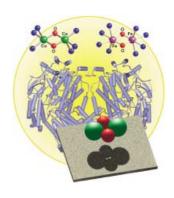
INTERNATION NAL EDITION

2002 41/7

Pages 1079-1254

COVER PICTURE

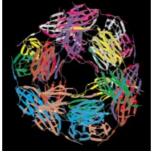
The cover picture shows $Cu_2(\mu-O)_2$ and $Fe_2(\mu-O)_2$ complexes with the $M_2(\mu-O)_2$ diamond core motif (the core is shown bottom right, M = green and oxygen = red spheres) and a representative example of a non-heme multimetal enzyme (hydroxylase component of methane monooxygenase, in the background). Although quite a familiar feature in high-valent manganese chemistry, the $M_2(\mu-O)_2$ diamond core motif has only recently been found in synthetic complexes for M = Cu or Fe. Despite differences in electronic structures that have been revealed through experimental and theoretical studies, $Cu_2(\mu-O)_2$ and Fe₂(μ-O)₂ cores exhibit analogously covalent metal–oxo bonding, and similar tendencies to abstract hydrogen atoms from substrates. Our understanding of biocatalysis has been enhanced significantly through the isolation and comprehensive characterization of the $Cu_2(\mu-O)_2$ and $Fe_2(\mu-O)_2$ complexes. In particular, it has led to the development of new mechanistic notions about how non-heme multimetal enzymes, such as, methane monooxygenase, fatty acid desaturase, and tyrosinase, may function in the activation of dioxygen to catalyze a diverse array of organic transformations. To find out more see the review by L. Que, Jr. and W. B. Tolman on p.1114 ff.



REVIEWS

-Contents

A complex and sophisticated machinery of proteins is involved in cellular protein folding and allows the functional state of proteins to be maintained under conditions in which they would normally unfold and aggregate. The structure of such a system, the small heat-shock protein from the archaeon *Methanococcos jannaschii*, is depicted. The principle features of these molecular chaperones, their structure – activity relationships, and the underlying molecular mechanisms are discussed in this review.



Angew. Chem. 2002, 114, 1142-1158

Keywords: chaperone proteins • protein aggregation • protein folding • protein structures • structure – activity relationships

S. Walter, J. Buchner* 1098–1113

Molecular Chaperones—Cellular

Machines for Protein Folding

Diamonds with iron and copper:

The recent characterization of metastable iron and copper complexes with the diamond-shaped bis(μ -oxo) core (see picture) has provided new insights into dioxygen activation and catalysis at dimetal sites in proteins. Recent detailed studies have shown that the iron and copper compounds



have divergent electronic structures, yet exhibit a number of related structural features, spectroscopic properties, and modes of reactivity.

Angew. Chem. 2002, 114, 1160-1185



L. Que, Jr.,* W. B. Tolman* . 1114-1137

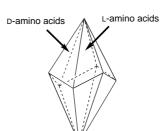
 $Bis(\mu$ -oxo)dimetal "Diamond" Cores in Copper and Iron Complexes Relevant to Biocatalysis

Keywords: bioinorganic catalysis • copper · iron · metalloenzymes · metal-oxo complexes · oxygen activation

MINIREVIEW:

Chirality existed before hands developed: Molecular chirality constitutes a fascinating topic linked to the origin of life itself. This minireview highlights some of the recent developments in this area which in part rediscover past reflections and achievements, and approaches the question whether the chirality of living systems arises from subtle quantum-electroweak force effects or from a more familiar chemistry, specifically the chiroselective adsorption of some amino acids onto minerals with enantiomeric faces (see picture).

Angew. Chem. 2002, 114, 1187-1193



Chirality of Living Systems: A Helping Hand from Crystals and Oligopeptides

Keywords: autocatalysis • biomineralization • chirality • origin of life • prebiotic chemistry



The following communications are "Very Important Papers" in the opinion of two referees. They will be published shortly (those marked with a diamond will be published in the next issue). Short summaries of these articles can be found on the Angewandte Chemie homepage at the address http://www.angewandte.com

Semiconductor Nanohelices Templated by Supramolecular Ribbons

E. D. Sone, E. R. Zubarev, S. I. Stupp*

Phase-Sensitive Supramolecular Chirogenesis in Bisporphyrin Systems

V. V. Borovkov,* T. Harada, Y. Inoue, R. Kuroda*

The Stable Pentamethylcyclopentadienyl Cation

J. B. Lambert,* L. Lin, V. Rassolov

Polymeric Surfactants for the New Millennium: A pH-Responsive, Zwitterionic, Schizophrenic Diblock Copolymer

S. Liu, S. P. Armes*

Cyclo[8]pyrrole: A Simple-to-Make Expanded Porphyrin with No Meso Bridges

D. Seidel, V. Lynch, J. L. Sessler*

Cyclotetrasilenide Ion: A Reversible Redox System of Cyclotetrasilenyl Cation, Radical, and Anion

T. Matsuno, M. Ichinohe, A. Sekiguchi*

Synthesis and Structure of Na₃N

D. Fischer, M. Jansen*

CORRESPONDENCE



A reanalysis of the results obtained by Cossío and de Lera et al. show that reactions A and B (see scheme) are essentially pericyclic even though they are favored by the electron lone pair on the heteroatom. This lone pair provides a reaction path with a decreased energy investment but the essential features of the process are typical of a disrotatory electrocyclization as in reaction C.

$$\langle x \rangle \rightarrow \langle x \rangle$$

X = NH reaction A X = O reaction B $X = CH_2$ reaction C J. Rodríguez-Otero,* E. M. Cabaleiro-Lago 1147 – 1150

Electrocyclization of (Z)-1,2,4,6-Heptatetraene and its Heterosubstituted Analogues: Pericyclic or Pseudopericyclic?

Angew. Chem. 2002, 114, 1195-1198

Different assumptions and considerations used in the calculations of Rodríguez-Otero and Cabaleiro-Lago account for the different interpretations of the mechanism for reactions A and B (see scheme). However, the authors believe that geometric, electronic, orbital, and magnetic criteria indicate that reactions A and B take place by a pseudopericyclic mechanism, whereas the all-carbon equivalent (reaction C) corresponds to a disrotatory (aromatic) electrocyclization.

Angew. Chem. 2002, 114, 1198-1200

A. R. de Lera, F. P. Cossío* . 1150–1152

Keywords: ab initio calculations • aromaticity • density functional calculations • electrocyclic reactions • transition states

COMMUNICATIONS

Immobilized gold nanoparticles on a three-dimensionally ordered macroporous (3DOM) film were prepared by a dipping method. The localized surface plasmon resonance of the nanoparticles and the stop-band of the 3DOM film, both still evident after immobilization, are both dependent on the refractive index of the surrounding medium. The picture shows a 3DOM film before (left) and after (right) coating with gold nanoparticles.

Angew. Chem. 2002, 114, 1201-1204



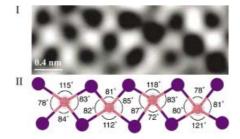
Z.-Z. Gu, R. Horie, S. Kubo, Y. Yamada, A. Fujishima, O. Sato* 1153 – 1156

Fabrication of a Metal-Coated Three-Dimensionally Ordered Macroporous Film and its Application as a Refractive Index Sensor

Keywords: colloids • gold • nanostructures • sensors • UV/Vis spectroscopy

Unknown in bulk BaI_2 : Ba coordination numbers of 5 and 6 are present in the one-dimensional (1D) crystal chains formed by BaI_2 within single-walled carbon nanotubes (SWNTs). This is revealed by high-resolution transmission electron microscopy images (I). The derived structure model II shows the bond angles measured for equatorial BaI_4 units along the central 1D octahedral chain within the BaI_2 chain in an SWNT capillary.

Angew. Chem. 2002, 114, 1204-1207



A One-Dimensional BaI₂ Chain with Five- and Six-Coordination, Formed within a Single-Walled Carbon Nanotube

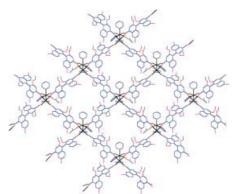
Keywords: barium • electron microscopy • halides • nanotubes



A chiral two-dimensional network is

the basis for the structure of a homochiral solid exploiting metalcarboxylate coordination. The synthesis involved enantiopure bridging ligands and metal-organic secondary building units (SBUs), and resulted in ethoxy-protected BI-NOL functionalities pointing into the cavities in this crystalline chiral zeolitic material (see picture).

Angew. Chem. 2002, 114, 1207-1210

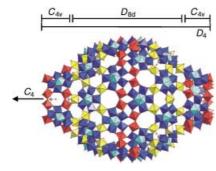


Y. Cui, O. R. Evans, H. L. Ngo, P. S. White, W. Lin* 1159–1162

Rational Design of Homochiral Solids Based on Two-Dimensional Metal Carboxylates

Keywords: chirality • coordination polymers · crystal engineering · supramolecular chemistry · zeolite analogues

Molybdenum monsters: A huge molybdenum cluster, comprising large areas with different local symmetry (in the picture different colors represent different (smaller) Mo fragments) is prepared and characterized. Molybdate fragments, which can tolerate a change of electron density at various positions, can be linked in a tremendous variety of ways according to a split-and-link process. Small changes of the boun-



A. Müller,* E. Beckmann, H. Bögge, M. Schmidtmann, A. Dress . 1162-1167

Inorganic Chemistry Goes Protein Size: A Mo₃₆₈ Nano-Hedgehog Initiating Nanochemistry by Symmetry Breaking

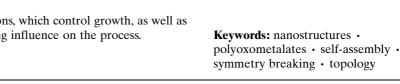
dary conditions, especially the redox conditions, which control growth, as well as the H⁺ ion concentration, can have a deciding influence on the process.

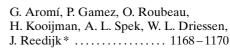
Angew. Chem. 2002, 114, 1210-1215

A space-ship-like Cu₈ cluster (see picture) results from the linking of two linear [Cu^{II}] units previously assembled through the template action of a pentadentate O ligand. The core of the cluster represents an entirely new topology within the vast family of magnetically coupled

Cu aggregates. The alkoxide-bridged octanuclear units are connected to each other by nitrate ligands into an unprecedented cluster polymer.

Angew. Chem. 2002, 114, 1216-1218



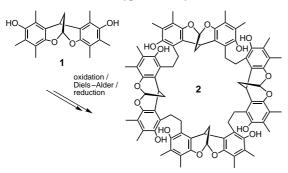


Ligand-Templated Four-Metal Chains Dimerize into a Unique [CuII8] Cluster

Keywords: cluster compounds • copper • magnetochemistry · supramolecular chemistry · template effect



It might not be as healthy as vitamin E, but the α -tocopherol derivative 1 undergoes all the typical reactions of the vitamin twice, as a result of its "siamese twin" character. The transformation of 1 in a three-step reaction sequence of oxidation, hetero-Diels - Alder reaction, and reduction provides facile access to tetrameric calixarene-type macrocycles 2.



Angew. Chem. 2002, 114, 1219-1221

T. Rosenau, A. Potthast, A. Hofinger,

Calixarene-Type Macrocycles by Oxidation of Phenols Related to Vitamin E

Keywords: cycloaddition • Diels – Alder reaction · macrocycles · spiro compounds · vitamin E

Blood and lymph vessel growth is regulated by Tie-2 and the VEGFR-3 receptor tyrosine kinases. These proteins also play major roles in the growth and metastasis of cancers. A novel class of inhibitors of these signal-transducing proteins, and of the IGF1R kinase (such as 1), is identified from a natural product derived compound library. This discovery opens up new opportunities for the development of antitumor agents.

P. Stahl, L. Kissau, R. Mazitschek, A. Giannis,* H. Waldmann* 1174–1178

Natural Product Derived Receptor Tyrosine Kinase Inhibitors: Identification of IGF1R, Tie-2, and VEGFR-3 Inhibitors

Keywords: angiogenesis • antitumor agents • combinatorial chemistry • natural products • tyrosine kinases

Kinetic inertness overcome: The selective coupling of Ge–E bonds (E = halogen, C) can be achieved at 130°C by direct synthesis from activated germanium. Alkyl bromides with an additional carbonyl function (see scheme) can be converted in a controlled manner into dibromodiorganogermanes.

Angew. Chem. 2002, 114, 1237-1239

S. Schlecht* 1178-1180

Selective Direct Synthesis of Organofunctionalized Dialkylgermanes from Solvochemically Activated Germanium

Keywords: amorphous materials • direct synthesis • germanium • main group elements

Adjustable belts: Selective synthesis of beltlike cyclophanes containing organometallic π systems is now possible. Intra- and intermolecular ring closure of the flexible ten-membered ring units of 1 leads to tetramer 2 and octamer 3, respectively (see picture; cod = cyclooctadiene).

Angew. Chem. 2002, 114, 1231-1234

Beltlike Macrocycles with Four and Eight (Cyclopentadienyl)(cyclobutadienyl)-cobalt Building Blocks, Formed by Stepwise Oligomerization of 1,6-Cyclodecadiyne Units

Keywords: alkynes \cdot cobalt \cdot cycloaddition \cdot cyclophanes \cdot macrocycles

A further component for molecular electronics: A single molecule insulator is realized by immobilizing a platinum(II) complex between two gold electrodes (see picture). The current/voltage characteristics of the setup shows the typical behavior of an insulator with a tunnel barrier height of 2.5 eV.

A *trans*-Platinum(II) Complex as a Single-Molecule Insulator

Keywords: electron transport • insulators • molecular electronics • platinum • single-molecule studies

Terminal addition of an electrophilic phosphanediyl complex to the nitrile-nitrogen atom of a Wittig ylide yields the nitrilium phosphane ylide complex **I** as an intermediate. Intramolecularly trapping of **I** by a

[3+2] cycloaddition with a *P*-phenyl group leads to a bowl-shaped, coordinatively bonded, novel tricycle.

Angew. Chem. 2002, 114, 1226-1228

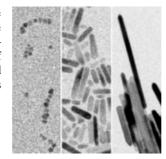
Intramolecular [3+2] Cycloaddition of a Nitrilium Phosphane Ylide Complex to the *P*-Phenyl Group of a Wittig Ylide

Keywords: cycloaddition • 1,3-dipoles • nitrogen heterocycles • phosphorus heterocycles • ylides



Oriented attachment is the key: Single crystalline ZnO nanorods with lengths up to 500 nm could be prepared in a stepwise manner from quasi-spherical nanoparticles. Only after the formation of pearl-chain-like structures (left), do the aggregated particles fuse upon heating to form nanorods (center and right).

Angew. Chem. 2002, 114, 1234-1237



Self-Assembly of ZnO: From Nanodots to Nanorods

Keywords: colloids • crystal growth • nanostructures • oxides • zinc

The electronic and steric properties of the 2,6-bis(trifluoromethyl)phenyl (Ar_f) substituent stabilize a naked dicoordinate phosphorus anion (see scheme) in the primary phosphanide salt $[K([15]crown-5)_2][Ar_fPH]$, both in the solid state and in solution.

V. L. Rudzevich, H. Gornitzka,

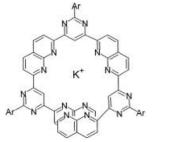
K. Miqueu, J.-M. Sotiropoulos, G. Pfister-Guillouzo, V. D. Romanenko,

The First "Naked" Primary Phosphanide Anion [ArPH]

Keywords: acidity \cdot anions \cdot coordination modes \cdot phosphanides \cdot phosphorus

Angew. Chem. 2002, 114, 1241-1243

Alternating naphthyridine – pyrimidine strands self-organize into a helical conformation containing a strongly polar cavity. The binding of metal and ammonium cations promotes supramolecular self-assembly in a sort of effector-induced growth process to form polymolecular stacks (see schematic representation) and fibers observable by electron microscopy. Such entities offer features of molecular ion channels.





Angew. Chem. 2002, 114, 1243-1246

Cation-Promoted Hierarchical Formation of Supramolecular Assemblies of Self-Organized Helical Molecular Components

Keywords: cations • helical structures • heterocycles • self-assembly • supramolecular chemistry

Chirality resolved: The stereochemistry of potential intermediates in the enzymic synthesis of 6-methyl salicylic acid, (R)-1 and (S)-1, has been determined by X-ray crystallography of two corresponding crystalline intermediates (S,S)-2 (shown) and (S,S)-3 (not shown).

Angew. Chem. 2002, 114, 1246-1250

K.-M. Cheung, S. J. Coles, M. B. Hursthouse, N. I. Johnson, P. M. Shoolingin-Jordan* ... 1198–1202

The Determination of the Absolute Configurations of Diastereomers of (*S*)-Camphanoyl 3-Hydroxy-5-oxohexanoic Acid Derivatives by X-ray Crystallography

Keywords: chiral auxillaries • configuration determination • enzymes • polyketides • X-ray diffraction

Stabilization of a peroxocarbonate ligand by formation of a five-membered chelate ring. The mononuclear peroxocarbonate complex **1** was prepared by the reaction of a bis(μ -hydroxo)diiron(III) complex with H_2O_2 and CO_2 . Compound **1** is the first crystallographically characterized transition metal complex with a peroxocarbonate ligand. Formation of the peroxocarbonate moiety in **1** proceeds by a nucleophilic addition of a peroxide anion to CO_2 . Hqn = quinaldic acid.

$$Fe^{3+} O Fe^{3+} DMF, -60°C$$

$$[Fe_2(qn)_4(OH)_2] \cdot 2H_2O$$

Angew. Chem. 2002, 114, 1250-1253

A New Mononuclear Iron(III) Complex Containing a Peroxocarbonate Ligand

Keywords: iron • oxygen activation • peroxo ligands • Raman spectroscopy • X-ray diffraction

Highly efficient chirality transfer is employed in controlling the topological asymmetry of chiral cages with propeller-like shape and pseudo C_3 symmetry (see picture; R = H, Cl). The cages are formed when two tripodal units (a tribenzylamine and a tris(tertiary phosphane)) become linked by three P=N bonds, by means of a tripod-tripod coupling process followed by a remarkable triple expulsion of molecular nitrogen.

Angew. Chem. 2002, 114, 1253-1256

R CH₃

Helical Sense Bias Induced by Point Chirality in Cage Compounds

Keywords: cage compounds · chirality · cooperative effects · helical structures · phosphazenes

Oxidation favors oxidative addition: This unexpected behavior has been observed in d⁸d⁸ 'open-book' diiridium complexes with alkene ligands, which become C–H activating after oxidation with ferrocinium salts (see scheme).

Angew. Chem. 2002, 114, 1256-1259

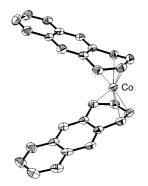
M. V. Jiménez, E. Sola, J. Caballero, F. J. Lahoz, L. A. Oro* 1208–1211

Alkene C-H Activations at Dinuclear Complexes Promoted by Oxidation

Keywords: C—H activation • hydride ligands • iridium • metal – metal interactions • oxidation

Two remarkable features of bis(anthracene)-cobaltate(1-) (see picture) are that it is the first homoleptic anthracene transition metal complex to be prepared by a conventional synthesis, that is, one not involving a metal atom reactor, and it is the first anionic complex of a late (Groups 7-11) transition metal containing only bound aromatic hydrocarbons.

Angew. Chem. 2002, 114, 1259-1263



W. W. Brennessel, V. G. Young, Jr., J. E. Ellis* 1211 – 1215

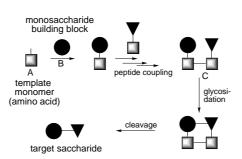
Bis(1,2,3,4- η^4 -anthracene)cobaltate(1 –)

Keywords: arene ligands \cdot cobalt \cdot diene ligands \cdot isocyanide ligands \cdot phosphane ligands



"Zipping up" a carbohydrate can be as simple as glycosidation of sugar units that are attached to a solid-phase bound peptide framework (see schematic presentation). The target disaccharides can be prepared efficiently, and the peptide template was found to influence the stereoselectivity of the glycosidation process.

Angew. Chem. 2002, 114, 1263-1266



D. R. Greenwell, A. F. Ibnouzaki, S. L. Warriner* 1215–1218

Peptide-Templated Saccharide Synthesis on a Solid Support

Keywords: carbohydrates • peptides • solid-phase synthesis • synthetic methods • template synthesis

The size of the ring formed in the Rh^I-catalyzed intramolecular hydroacylation of 4,6-dienals is dependent on the presence of a substituent at C7 and the geometry of

Cycloheptenones by RhI-Catalyzed

Intramolecular Hydroacylation of

Y. Sato, Y. Oonishi,

4,6-Dienals

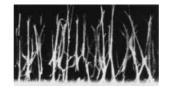
the olefin. Cyclization of 4,6-dienals with a substituent at the C7 position (1; R^1 = alkyl, R^2 = H) produced cycloheptenones (2) as the major product (see scheme), while that of 4,6-dienals with no substituent at the terminus of the diene (1; R^1 = H) preferentially gave cyclopentanones. The cyclization of substrates containing a 6E olefin produced cycloheptenones as the major product, while a cyclopentanone was obtained from a substrate having a 6Z olefin. dppe = 1,2-bis(diphenyl-phosphanyl)ethane.

Keywords: aldehydes • cyclization • dienes • hydroacylation • rhodium

Angew. Chem. 2002, 114, 1266-1269

A water contact angle exceeding 170° is exhibited by the surface of aligned polyacrylonitrile (PAN) nanofibers without any surface treatment (the picture shows a cross-sectional view of the assynthesized nanofibers). The nanofibers were obtained by simply extruding a PAN solution through an anodic alumina template into a solidifying solution. The factors that govern the hydrophobicity of aligned nanostructures are discussed.

Angew. Chem. 2002, 114, 1269-1271

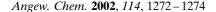


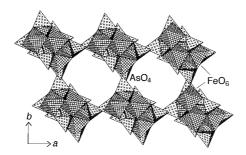
L. Feng, S. Li, H. Li, J. Zhai, Y. Song, L. Jiang,* D. Zhu 1221 – 1223

Super-Hydrophobic Surface of Aligned Polyacrylonitrile Nanofibers

Keywords: electron microscopy • hydrophobic effect • nanostructures • surface chemistry

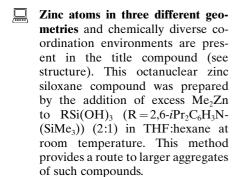
Three-dimensions from one: One-dimensional iron arsenate oxalate is prepared, and shown to be reactive, giving rise to a two- and a mixed-valent (Fe^{III}/Fe^{II}) three-dimensional iron arsenate structure (see picture). The sequential crystallization provides evidence for the building up of open architectures from low to higher dimensional structures, demonstrating the "life of a chemical system".



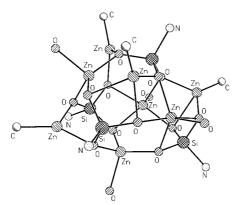


A Reactive Intermediate in the Synthesis of Iron Arsenates: Synthesis of the First One-Dimensional Iron Arsenate Oxalate and Its Transformation into Two- and Three-Dimensional Iron Arsenates

Keywords: crystal growth • iron • microporous materials • mixed-valent compounds • solid-state structures



Angew. Chem. 2002, 114, 1274-1277

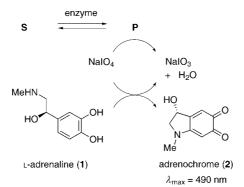


[Zn₄(thf)₄(MeZn)₄(O₃SiR)₄] (R = 2,6iPr₂C₆H₃N(SiMe₃)), A Compound Containing Trigonal-Planar, Tetrahedral, and Trigonal-Bipyramidal Metal Atoms: A New Route to Larger Aggregates

Keywords: aggregation • cage compounds • silicon • zinc

A versatile high-throughput enzyme assay is demonstrated which is based on the colorimetric back-titration of sodium periodate with Ladrenaline (1). Enzyme activity is associated with the depletion of sodium periodate by the reaction product (P), and indicated by the decrease in the amount of the red dye adrenochrome (2) produced by the oxidation of adrenaline by sodium periodate.

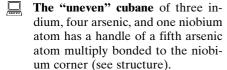
Angew. Chem. 2002, 114, 1277-1280



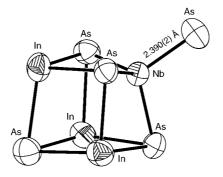
D. Wahler, J.-L. Reymond* . 1229 – 1232

The Adrenaline Test for Enzymes

Keywords: enzyme assays • high-throughput screening • hydrolases • lipases • phytases



Angew. Chem. **2002**, 114, 1280–1282



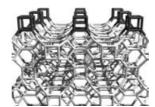
F. Gascoin, S. C. Sevov* 1232 – 1234

Cubane with a Handle: $[\{In_3As_4Nb\}-As\}^{7-} \text{ in } Cs_7NbIn_3As_5$

Keywords: arsenic • multiple bonds • polyanions • solid-state structures • Zintl phases



New insights into crystal growth for zeolite Beta C are shown from simulation studies. Crystal growth may be facilitated by double 4-rings (see picture), giving rise to two distinct external surface topologies. The predictions concur with and explain recent experimental findings and provide evidence that the nanoscopic surface structures arise from complex reactions of siliceous oligomers with the zeolite surface.



Surface Structure and Crystal Growth of Zeolite Beta C

Angew. Chem. 2002, 114, 1283-1285

Keywords: computer chemistry • crystal growth • surface chemistry • zeolites



Easy and clean: A palladium(0)-catalyzed decarbonylative Heck olefination of activated esters of aryl, heteroaryl, and vinyl carboxylic acids opens up an opportunity for a novel waste-free Heck olefination process (see scheme).

Angew. Chem. 2002, 114, 1285-1289

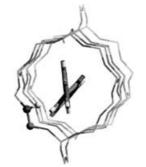
L. J. Goossen,* J. Paetzold . . 1237 – 1241

Pd-Catalyzed Decarbonylative Olefination of Aryl Esters: Towards a Waste-Free Heck Reaction

Keywords: arenes • esters • Heck reaction • homogeneous catalysis • palladium



Stabilization of electron-hole pairs for more than 12 months at room temperature was achieved by using biphenyl molecules occluded in the Al-ZSM-5 zeolite type (see picture). The unpaired electrons are localized in the zeolite framework in the vicinity of the occluded biphenyl molecule. Results establish that the trapped electron-hole pair acquires a ferromagnetic ground state.



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Electron-Hole Pairs Stabilized in Al-ZSM-5 Zeolites

Keywords: biphenyl • electron transfer • EPR spectroscopy • Raman spectroscopy • zeolites



Supporting information on the WWW

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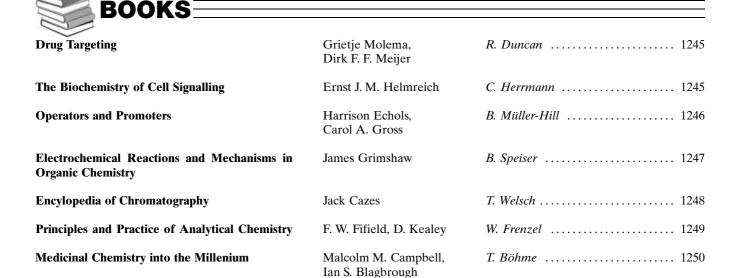


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